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PRELIMINARY SELECTION OF COMPATIBLE SOLVENTS FOR VINYL PAINTS.(U)
MAR 79 A BEITELMAN, R LAMPO

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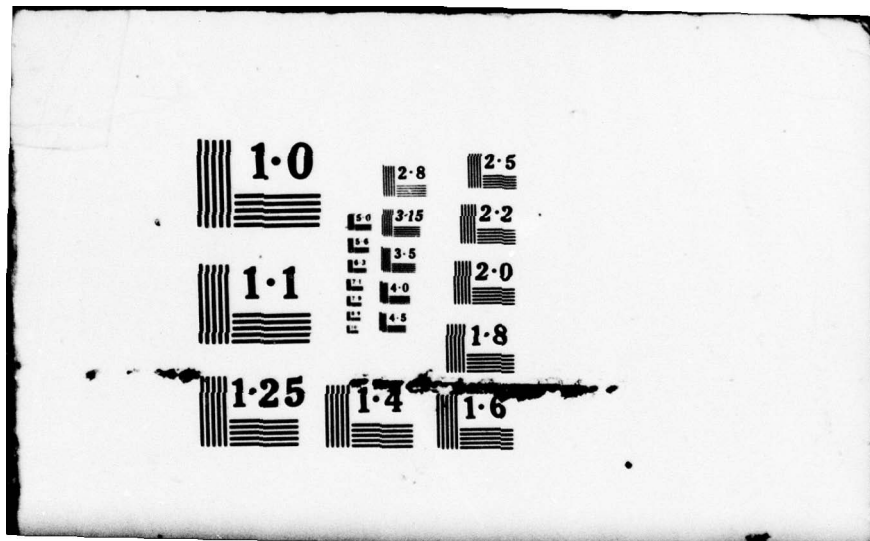
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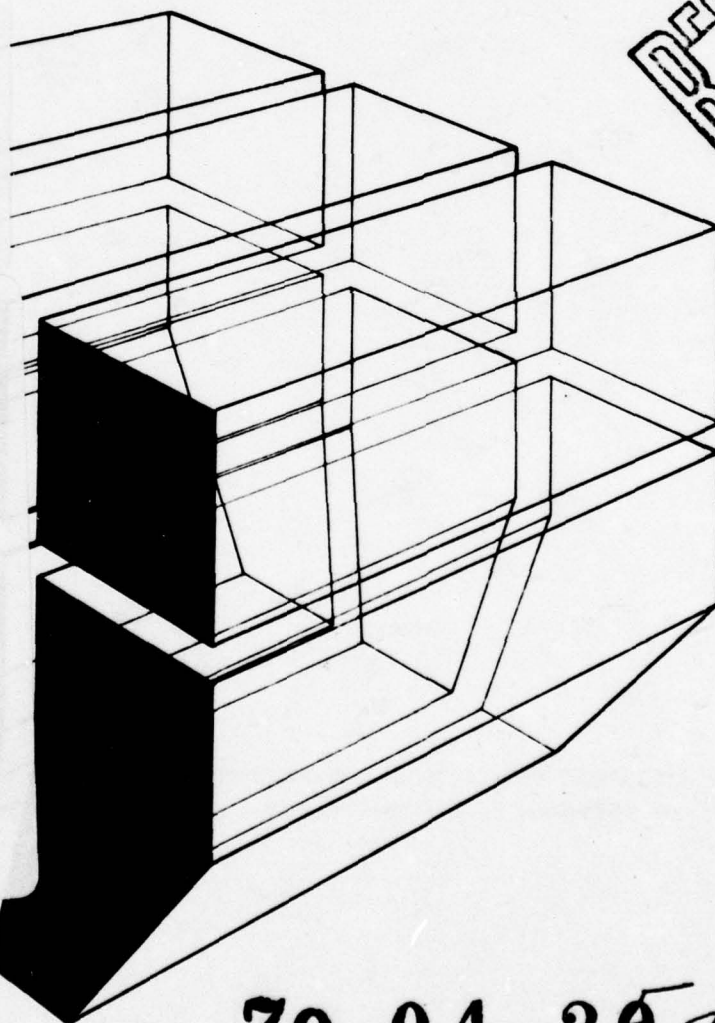
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PRELIMINARY SELECTION OF COMPATIBLE
SOLVENTS FOR VINYL PAINTS

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Alfred Beitelman
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Block 20 continued.

a vinyl formulation was used. The viscosity, water permeability, tensile strength, and retained solvents of this new formulation were compared with existing vinyl formulations. It was concluded that although the new formulation has properties very similar to the existing formulations, the MAK must be stabilized for the coating to have an acceptable shelf life.

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FOREWORD

This research was performed for the Directorate of Civil Works, Office of the Chief of Engineers (OCE), by the Engineering and Materials (EM) Division of the U.S. Army Construction Engineering Research Laboratory (CERL), under Civil Works Research Work Unit 31205. The OCE Technical Monitor was Mr. L. G. Guthrie, DAEN-CWE-DS.

Dr. G. R. Williamson is Chief of EM. COL J. E. Hays is Commander and Director of CERL, and L. R. Shaffer is Technical Director.

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PRELIMINARY SELECTION OF COMPATIBLE SOLVENTS FOR VINYL PAINTS

1 INTRODUCTION

Background

For many years, the basic paint system used by the Corps of Engineers on locks and dams along inland waterways has been based on vinyl resins. The initial formulation has undergone many changes since it was developed in 1947 in order to make use of more durable pigments, more effective plasticizers, and alternate solvents. However, most changes within the past 8 years have been to modify the formulation solvent system to comply with safety and air pollution regulations.

The most recent Civil Works Guide Specification CW-09940, Painting: Hydraulic Structures and Appurtenant Works (Department of the Army, January 1977) contains the vinyl formulations currently used by the Corps. Nine formulations are listed including a zinc primer; red, white, and gray general purpose paints; black and aluminum topcoat paints; and a high-build additive package material. Also listed are six thinners which must be used with the appropriate vinyl paints at various ambient temperatures.

CW-09940 imposes several limits on the use of these vinyl paints as paint systems because air pollution regulations enacted in many states restrict the use of photochemically reactive solvents. As a result of these restrictions, some paints are formulated with a large percentage of a nonphotochemically-reactive nitropropane solvent. Although this solvent is compatible with vinyl resins, it creates adhesion problems when used with the vinyl zinc primer. As a result, neither the vinyl zinc primer nor the high-build additive package which requires the zinc-rich primer can be used in states with restrictive air pollution regulations.

In states where the restrictive air pollution regulations are not in effect, paints containing aromatic solvents and branch chained ketones can be used. These solvents are compatible with the zinc-rich primer and are less expensive than the nitropropane solvent.

This dual set of paint formulations -- one to meet air pollution regulations, the other to employ the zinc primer and reduce cost -- has created problems both for design engineers and maintenance units. The engineer's problem is one of selecting an appropriate paint system for the structure. Since at the time of the design it may not be known in

which state the painting operation will take place, the engineer may be forced to select paints containing nitropropane solvents, thereby needlessly sacrificing both a superior system and cost benefits. Maintenance units, on the other hand, must maintain dual supplies of paints. If supplies are intermixed by chance and a topcoat containing nitropropane solvent is applied over a vinyl zinc primer, a system with an expected life of 20 to 40 years will probably fail within several weeks.

Objective

The overall objective of this study is to produce a single set of vinyl paint formulations for use within the Corps of Engineers that meet current state air pollution regulations and are compatible with the vinyl zinc primer.

The objective of this report is to document the selection of solvents which are (1) potentially compatible with standard coatings, and (2) meet air pollution regulations.

Approach

Numerous solvents and solvent systems were screened by incorporating the solvent or solvent system into a small batch of paint. The paint was then applied to steel panels and the panels exposed to tap water for several weeks. The paints were subjectively evaluated as to their application properties, viscosity, and adhesion to steel and to the vinyl zinc primer. The most promising solvents were then incorporated into a paint formulation. Physical properties of this formulation were compared to those of paint formulations currently used by the Corps of Engineers.

Mode of Technology Transfer

Formulations developed as a result of this study will be incorporated into Civil Works Guide Specification CW 09940, Painting: Hydraulic Structures and Appurtenant Works.

2 SCREENING PROGRAM

General

All coatings were made in the laboratory. As a starting point, the formulas V-766e and VZ-108d listed in CW-09940 were modified by replacing the specified solvent systems with the new solvents being evaluated. (See Appendix A.) Solvents were selected for screening based on their potential solubility for vinyl resins, their evaporation rate, and their regulated use under current air pollution laws. They included ethyl propyl ketone methyl amyl ketone (MAK), ethyl amyl ketone, methoxy acetone, cyclohexanone, isobutyl acetate, n-butyl acetate, ethylene glycol monoethyl ether acetate, and ethylene glycol monomethyl ether. (Ethylene glycol monomethyl ether was later eliminated from the program when it was learned that it may be absorbed through the skin in toxic amounts.)

Screening Results

The screening program showed that although acetates are good solvents for vinyls, they have adverse effects on the performance of the vinyl zinc primer. These effects were similar to those observed when nitropropane solvent is used, i.e., poor adhesion, blistering, and film embrittlement after several weeks of immersion in tap water. Samples of ethyl amyl ketone and methoxy acetone contained excessive amounts of acidity which produced poor adhesion of the V-766 formulation. (All ketones break down to form acids over a period of time; however, this reaction is more rapid with some ketones than others.) The samples of both the ethyl amyl ketone and the methoxy acetone were fresh, yet excessively acidic when received. Thus, they were judged to be unusable for Corps vinyl coatings.

Cyclohexanone caused the film to remain wet for an excessive length of time. V-766e has been formulated such that numerous coats may be applied in succession allowing very little drying between coats. This rapid drying allows contractors to apply a complete paint system in 1 day. Moreover, the rapid-evaporating solvents allow the coating to attain a high degree of abrasion resistance within a short time. The use of cyclohexanone would adversely affect both of these properties, and therefore, it was eliminated from the program.

Methyl propyl ketone (MPK) and MAK were both found to be good solvents for vinyl resin. Fresh samples had low acidity and were compatible with both the regular vinyl resin and the vinyl zinc primer coatings. It was decided that these two solvents would be blended to produce a relative evaporation rate approximately equal to that of the

solvents in V-766e, and that the performance qualities of a coating made with this solvent blend would be compared with coatings specified in CW-09940.

MPK and MAK Blend

MPK and MAK were combined in a vinyl paint similar to V-766e. The combined evaporation rate was adjusted to produce acceptable spraying properties. The total solids were adjusted to produce a viscosity similar to V-766e. The resulting formula is listed in Table 1.

Table 1
Formula No. 1253

<u>Material</u>	<u>Percent by Weight</u>
Vinyl resin type 4 ¹	12.2
Vinyl resin type 3 ²	6.1
Diisodecyl phthalate	3.1
Titanium dioxide ³	14.0
MAK	20.6
MPK	32.2
Toluene	11.6
Ortho-phosphoric acid	0.2
	<hr/> 100.0

¹ Union Carbide Vinylite VMCH

² Union Carbide Vinylite VYHH

³ Titanium Pigment Corporation Titanox 2062

A laboratory batch of this formulation was made by charging all the raw materials, except the phosphoric acid and a small amount of the MPK, into a gallon-size pebble mill jar and milling for 3 days. After removal from the pebble mill, the material was placed on a high-speed stirrer. The ortho-phosphoric acid was diluted with 10 volumes of MPK and added slowly as the material was being agitated.

Formula No. 1253 was evaluated in side-by-side tests with V-766e and V-766e (AP). (See Appendix A for specification requirements.) To insure uniformity, these specification materials were also made up in the laboratory using the same techniques employed in the manufacture of formulation No. 1253.

Tests Performed

Acidity

The acidity of the solvents was tested according to Federal Test Method Standard No. 141a, Method 5252. Results were recorded as milligrams of potassium hydroxide required to neutralize 1 gram of the sample solvent.

Viscosity

The viscosities of the paints before and after thinning were checked using a No. 4 Ford cup in accordance with American Society for Testing and Materials (ASTM) test method D-1200.

Water Permeability

The relative water permeability of the coatings was observed using 25-cm Gardner-Park Permeability Cups and a test method similar to the one used by the New York Paint and Varnish Club and reported to the Federation Convention in 1937.¹ All coatings were spray applied to parchmentized paper to a dry film thickness of 5 mils. The applied coatings were dried for 1 day at 77°F (25°C), followed by 4 days at 120°F (48°C). The cups were assembled and placed in a desiccator containing anhydrous calcium sulfate such that the water was in contact with the paint. The desiccator was maintained at a constant temperature of 100°F (37°C). An initial weight was recorded after the cups had stabilized at the 100°F (37°C) temperature for 2 days and a final weight was taken 9 days later. Results were recorded as average weight loss per day for each cup. All samples were run in duplicate.

Tensile Strength

The relative tensile strength of the coatings was measured using a Gardner-Parks Tensile Strength Machine. All coatings were spray applied to photographic paper to a dry film thickness of 3.5 mils. The coatings were dried for 1 day at 77°F (25°C), followed by 4 days at 120°F (48°C). The films were then cut into 1-cm strips and removed from the photographic paper. Half the strips were placed in distilled water while the other half remained at laboratory conditions for 7 days before testing. Results were recorded as grams required to break the specimen.

¹ Payne, H. F., "Permeability Measurements," Paint and Varnish Proceedings, Scientific Section (National Paint, Varnish, and Lacquer Association, 1938).

Retained Solvents in Dried Coatings

Portions of each of the coatings were thinned with each of several present or potential thinners. After a period of days or weeks, samples of the dried coatings were analyzed for their solvent content using a gas chromatograph. The following technique was used: Samples were thinned 20 percent by volume with the appropriate thinner and drawn down with a doctor blade onto glass plates. The approximate dry film thickness was 5.5 mils. The coatings were dried at laboratory conditions. Samples of these coatings were removed from the glass plates for the solvent analysis after 1, 3, 8, 15, 30, and 57 days.

Samples of approximately 0.1 grams of the dried coating were placed into a test tube and weighed to the nearest 0.1 mg. One milliliter of acetone containing 0.11 percent benzene was added to the test tube and the material was reweighed. The test tube was shaken to dissolve the coating. Two milliliters of n-pentane were added to the test tube to remove the resin from solution. The test tube was reshaken and centrifuged.

The analyses were carried out using a Hewlett Packard Gas Chromatograph Model No. 5750 in the flame ionization (FID) mode. (See Table 2 for operating conditions.) The Tenax GC packing provided good separation of all but the MPK and benzene. The absence of MPK (see Appendix B) in any of the extraction was confirmed by using a 8-ft x 1/4-in. (2-m x 6-mm) ID column packed with 20 percent by weight Bentone 34 and diisodecylphthalate on 60/80 mesh WAW. The areas of the peaks were integrated using a Hewlett Packard recording integrator Model No. 3380A. Weight response factors relative to benzene were determined for each solvent used. Response factors are especially important with the nitropropane solvents which have a lower weight to response ratio than the other solvents.

The weight percent of each solvent remaining in the sample film was then determined:

1. The response for benzene was found by dividing the weight of the benzene used by the area of the benzene peak:

$$\frac{\text{Weight of benzene}}{\text{Area of benzene}} = \text{Benzene response}$$

2. The corrected area of each solvent was multiplied by the benzene response ratio to determine the weight of the solvent:

$$\text{Benzene response} \times \text{Area of solvent} = \text{Weight of solvent}$$

3. The weight percentage of each solvent remaining in the film was found by dividing the weight of the solvent by the weight of the sample film and multiplying by 100 percent:

$$\frac{\text{Weight of the Solvent}}{\text{Weight of the Sample Film}} \times 100\% = \text{Percent of solvent in film}$$

The results of these procedures are tabulated in Figure 1 and Appendix B.

Table 2

Gas Chromatograph Operating Conditions

Instrument: Hewlett Packard Model No. 5750 in flame ionization (FID) mode

Column: 10-ft x 1/4-in (3-m x 6-mm) ID stainless steel column packed with 60/80 mesh Tenax GC

Electrometer Range: 10^4

Carrier Gas: helium at 30 cc/minute

Injection Port Temperature: 325°C

Detector Block Temperature: 325°C

Column Temperature: 150 to 250°C at $20^{\circ}\text{C}/\text{minute}$ (1-minute post injection delay and 2-minute hold at end of programmed run).

Sample Size: approximately 5 μL

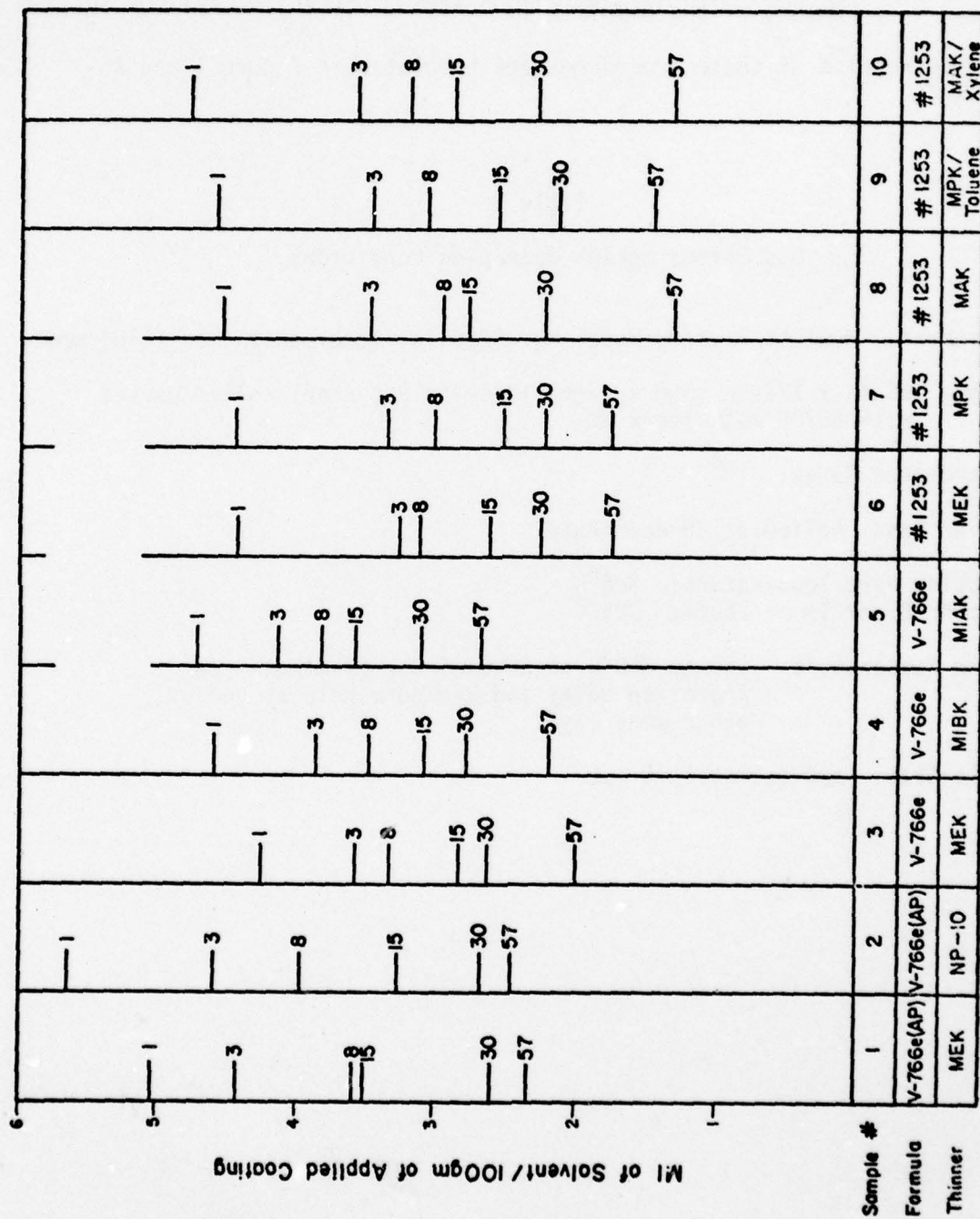


Figure 1. Total volume of solvent in 100 gm of applied coating.

3 TEST RESULTS AND ANALYSIS

As noted in Chapter 2, the acidity of ketone solvents often rises over a period of time. When CERL received the samples of MPK and MAK, they both had acidities of approximately 0.04 mg. After 1 year of storage, the MPK still had an acidity below 0.1 mg. However, the acidity of the MAK had increased to 0.4 mg. The acidity of a second sample of MAK purchased from a distributor had an initial acidity of 0.4, indicating, perhaps, that the material had aged at the distribution site. The current raw material requirements for methyl isoamyl ketone (MIAK) require the acidity to be less than 0.2 mg (See Appendix A). If it is to be assumed that this requirement for MIAK would be a reasonable requirement for MAK, it is obvious that the acidity is a problem which must be corrected before MAK could be specified for use in a Corps vinyl paint.

Table 3 shows the viscosity measurements of each of the formulations, along with the formulation thinned with 20 percent of specific thinners. It should be noted that the formulations were all based on a viscosity of about 75 to 80 seconds, and not on the solids content of the paint itself. When the percent volume solids in the formulations was calculated, it was determined that V-766e(AP) = 20.1; V-766e = 19.1; and No. 1253 = 20.3. Thus, it is apparent that formulation No. 1253, even with its slightly higher volume solids, has viscosity characteristics very similar to standard formulations.

The thinners used in conjunction with formulation No. 1253 were selected as potential thinners to be used at low, moderate, and high ambient temperatures. Ketone thinners were used because they met air pollution regulations. In addition, two thinners with large amounts of aromatic solvents were evaluated. The use of these thinners in states not having restrictive air pollution regulations would significantly reduce costs. All thinners were found to be compatible with the new formulation, and they reduced viscosity as efficiently as comparable thinners in standard formulations.

Table 4 shows the results of the water permeability test. Because these coatings are designed for immersion, this test was conducted with the water in direct contact with the surface of the coating; i.e., the cups were maintained in an inverted position. This is contrary to the ASTM test D-1653 for moisture vapor permeability which measures the vapor permeability of the coating. The test results of the duplicate samples, which were very reproducible, indicate that there is essentially no difference between the water permeability of the V-766e and Formula No. 1253. However, V-766e(AP) allowed less water to pass through the film.

Table 3
Viscosity Measurements

Sample Number	Formulation	Formulation Viscosity (sec)	Thinner Added	Viscosity of Thinned Material (sec)
1	V-766e(AP)*	77	MEK	35
2	"		NP-10*	37
3	V-766e*	77	MEK	37
4	"		MIBK	36
5	"		MIAC	38
6	No. 1253	79	MEK	34
7	"		MPK	35
8	"		MAK	40
9	"		9:1 toluene:MPK	37
10	"		9:1 xylene:MAK	37

* See Appendix A for specification requirements.

Table 4
Water Permeability Test Results

Sample Number	Formulation	Thinner	Average Daily Loss (gx10 ⁻⁴)
1	V-766e(AP)*	NP-10*	602
2	"	"	600
3	V-766e*	MIBK	699
4	"	"	689
5	No. 1253	9:1 toluene:MPK	700
6	"	"	693

*See Appendix A for specification requirements.

It was initially assumed that the amount of solvent remaining in the film -- either a greater or lesser amount of nitropropane -- caused the lower permeability of these samples. Gas chromatographic analysis, however, showed that the forced drying of the coatings had brought the total retained solvent levels of all three coatings to uniformly low levels in the 0.4 percent to 0.7 percent range.

Table 5 lists the results of the tensile strength test measurements. The strength of films was observed on similar films which were either dry or had been immersed in water for 1 week. Films which remained dry had consistently higher strength than their immersed counterparts, as expected. However, on both the wet and dry samples, films containing the nitropropane solvent had the greatest tensile strength. The ketone-containing films showed little variation with the exception of the wet sample of Formula No. 1253 thinned with MAK.

As with the water permeability test results, it was initially assumed that retained solvent may have affected these test results. Therefore, dry samples were analyzed for total solvent using a gas chromatograph. It was found that Samples 1, 2, 3, and 4 all had an equally low total solvent content -- in the range of 0.4 to 0.7 percent. Sample 5, however, retained a large amount of MAK, producing a total solvent content of 1.1 percent. This greater amount of retained solvent is sufficient to significantly affect the test results, and may be responsible for the single low tensile strength value.

Table 5
Tensile Strength

Sample Number	Formulation	Thinner	Tensile Strength Dry Sample (g)	Tensile Strength Wet Sample (g)
1	V-766e(AP)*	NP-10*	2660	2430
2	V-766e*	MIBK	2280	2230
3	No. 1253	9:1 toluene:MPK	2460	2130
4	"	MPK	2410	2110
5	"	MAK	2330	1870

*See Appendix A for specification requirements.

It should be pointed out that the reproducibility of this test method leaves much to be desired. ASTM has a tensile strength test method using automated application equipment and a load cell type tensile strength machine. The reproducibility of their method is only ± 5 percent. The method used in this test employed a standard hand-held spray gun and the Gardner-Park Tensile Strength Machine, which relied on

a spring and pointer to record the breaking strength of the specimen. Although repeated samples produced results within the +5 percent range, variations in application characteristics may have produced consistent variations in some samples.

The data obtained from evaluating the retained solvents in the dried coatings are shown in Appendix B both numerically and graphically on a weight basis for each individual solvent. Because of the difference in densities between the nitropropane solvent and the ketone solvents, the total percent solvent by weight was converted to total retained solvent by volume. These results are presented in Figure 1. The data in Figure 1 show:

1. Sample 6 was formulated and thinned as a potential low temperature replacement for both Samples 1 and 3. After the first day, this sample lost its solvent faster than the other two samples. Even on the first day, its retained solvent was at a lower level than that of the other paint meeting air pollution regulations (Sample 1).

2. Sample 7 was formulated and thinned as an all-purpose moderate temperature replacement for both Samples 2 and 4. In all cases, solvent release was greater from the experimental formulation than from the existing formulas.

3. Sample 8 was formulated and thinned as a high temperature replacement for both Samples 2 and 5. (Guide specification CW-09940 indicates that the material represented by Sample 2 may be used at both moderate and high temperatures.) In all cases, the solvent release from the experimental formulation was greater than from the existing formulations.

4. Samples 9 and 10 were thinned with low cost thinners which do not meet air pollution regulations. The use of these thinners could reduce the cost of painting in areas without air pollution regulations. When compared with Samples 4 and 5 (the materials currently used in these areas), the solvent release for each respective temperature range with the experimental formulation is equal to or greater than that produced when the existing formulations are used.

It should be noted that after a single day of air drying, neither MEK nor MPK could be detected in any of the samples. Conversely, in samples 6 through 10, MIBK -- an impurity in MPK -- was retained throughout the duration of the test.

4 CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The test results were analyzed and the following conclusions drawn:

1. If solvents MPK and MAK are combined, a test formulation paint can be produced which has a total solids content, initial viscosity, and thinning properties essentially equal to those of the formulations presently recommended in Civil Works Guide Specification CW-09940.

2. The test formulation paint has water permeability properties equal to the existing V-766e formulation. Although these permeability rates are slightly greater than that for V-766e(AP), a history of the comparative field performance of V-766e and V-766e(AP) indicates that the difference is inconsequential.

3. The test formulation paint has tensile strength properties essentially equal to the existing V-766e formulation. These properties are slightly inferior to those of V-766e(AP). This inferiority was previously unknown; its effect has not been noted in field service.

4. Retained solvent data show that the coating will release the solvents in the new formulation at a rate equal to or greater than the existing formulations. The superior solvent release will allow the new coating to attain its potential properties in a shorter time.

5. These individual tests confirm that the test formulation made with a combination of MPK and MAK has performance qualities essentially equal to the two formulations currently being used. However, continuation of acid stability observations initiated in the screening program shows that while MPK is very stable; the acidity of MAK tends to display a rapid increase. This increase is such that although fresh solvent is used to manufacture the paint, the paint probably would not meet the adhesion test after 1 year of storage. Unless this acidity build-up can be controlled, MAK cannot be used for the manufacture of vinyl paints.

Recommendations

The manufacturer of MAK has been informed of its instability and is attempting to locate a suitable MAK stabilizer. When a suitable stabilizer is found, the manufacturer will provide MAK as a stabilized product. At that time, the development and field testing of a full complement (V-766, V-102, V-103, V-106, and VZ-108) of vinyl coatings for use within the Corps of Engineers should be conducted.

APPENDIX A:

PAINT FORMULATIONS AND
FORMULATION SPECIFICATIONS*

Vinyl-Type White (or Gray) Paint

Formula V-766e

<u>Ingredients</u>	<u>Percent by Weight</u>
Vinyl resin, type 3	5.6
Vinyl resin, type 4	11.6
Titanium dioxide and (for gray) carbon black	13.0
Diisodecyl phthalate	2.9
Methyl isobutyl ketone	32.0
Toluene	34.7
Ortho-phosphoric acid	0.2
	<u>100.0</u>

Formula V-766e (AP)

<u>Ingredients</u>	<u>Percent by Weight</u>
Vinyl resin, type 3	5.4
Vinyl resin, type 4	11.1
Titanium dioxide and (for gray) carbon black	12.5
Diisodecyl phthalate	2.9
Toluene	11.2
Nitropropane solvent	48.0
Methyl ethyl ketone	8.7
Ortho-phosphoric acid	0.2
	<u>100.0</u>

*Information in Appendix A is drawn from Civil Works Guide Specification CW-09940, Painting: Hydraulic Structures and Appurtenant Works (Department of the Army [DA], January 1977).

Vinyl-Type Zinc-Rich Primer:

Formula VZ-108d

<u>Ingredients (Component A)</u>	<u>Percent by Weight</u>	<u>Pounds</u>	<u>Gallons</u>
Vinyl resin, type 3	16.6	109.2	9.65
Methyl isobutyl ketone	80.6	528.9	79.30
Suspending agent E	0.7	4.6	0.28
Suspending agent F	0.4	2.7	0.19
Methanol	0.5	3.3	0.50
Synthetic iron oxide (red)	1.2	7.9	0.19
	<u>100.0</u>	<u>656.6</u>	<u>90.11</u>

Ingredients (Component B)

Silane B	100.0	4.1	0.47
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Ingredients (Component C)

Zinc dust	100.0	550.0	9.42
			<u>100.00</u>
			(mixed paint)

Ingredient Materials and Thinners for Special Paint

Formulations

The following ingredient materials apply only to those paints whose formulations are shown above.

Carbon Black. Carbon black shall conform to Federal Specification TT-P-343.

Zinc Dust Pigment. Zinc dust pigment shall conform to Federal Specification TT-P-460, type I or II; paint to be used in potable water tanks or as otherwise directed shall contain type II pigment only.

Titanium Dioxide. The titanium dioxide in vinyl paint Formula V-766e shall be one of the following: Titanox 2060, 2061, or 2062, Titanium Pigment Corp.; Ti-Pure 960 or 966; E. I. DuPont DeNemours and Co., Inc.; Unitane OR-650, American Cyanamid Co.; Zopaque R-88S, Gliden Pigments.

Diisodecyl Phthalate. Diisodecyl phthalate shall have a purity of not less than 99.0 percent, shall contain not more than 0.1 percent water, and shall have an acidity (calculated as acetic acid) of not more than 0.005 percent by weight.

Vinyl Resin, Type 3. Vinyl resin, type 3, shall be a vinyl chloride-acetate copolymer of medium average molecular weight produced by a solution polymerization process and shall contain 85 to 88 percent vinyl chloride and 12 to 15 percent vinyl acetate by weight. The resin shall have film-forming properties and shall, in the specified formulations, produce results equal to "Vinylite" resin VYHH, as manufactured by the Union Carbide Corporation.

Vinyl Resin, Type 4. Vinyl resin, type 4, shall be a copolymer of the vinyl chloride-acetate type produced by a solution polymerization process, shall contain (by weight) 1 percent interpolymers of dibasic acid, 84 to 87 percent vinyl chloride, and 12 to 15 percent vinyl acetate. The resin shall have film-forming properties and shall, in the specified formulations, produce results equal to "Vinylite" resin VMCH, as manufactured by the Union Carbide Corporation.

Suspending Agent E. Suspending agent E shall be a light, cream-colored, finely divided powder having a specific gravity of 2 to 2.3. It shall be an organic derivative of magnesium aluminum silicate mineral capable of minimizing the tendency of zinc dust to settle firmly without increasing the viscosity of the paint appreciably; e.g., Bentone 14, produced by the Pigments and Chemical Div., NL Industries, Inc.

Suspending Agent F. Suspending agent F shall be a light, cream-colored, finely divided powder having a specific gravity of approximately 1.70. It shall be an organic derivative of a special montmorillonite; e.g., Bentone 27, produced by the Pigments and Chemical Div., NL Industries, Inc.

Orthophosphoric Acid. Orthophosphoric acid shall be a chemically pure, 85 percent grade.

Methanol (Methyl Alcohol). Methyl alcohol shall conform to ASTM Designation D 1152.

Methyl Ethyl Ketone (MEK). MEK shall conform to ASTM Designation D 740.

Methyl Isobutyl Ketone (MIBK). MIBK shall conform to Federal specification TT-M-268.

Methyl Isoamyl Ketone (MIAK). MIAK shall be at least 97 percent pure, shall have a distillation range of 139 to 149°C, and shall have specific gravity (at 20/20°C) of 0.812 to 0.815. The acidity in 1 gram of the material shall be neutralized by not more than .20 mg of potassium hydroxide.

Nitropropane Solvent. Nitropropane solvent shall have a distillation range of 119 to 133°C, shall have a specific gravity of .990 to .994 at 25/25°C, and shall have a maximum acidity of 0.2 percent (calculated as acetic acid); e.g., Nipar S-30, manufactured by Commercial Solvents Corp.

Toluene. Toluene shall conform to Federal Specifications TT-T-548.

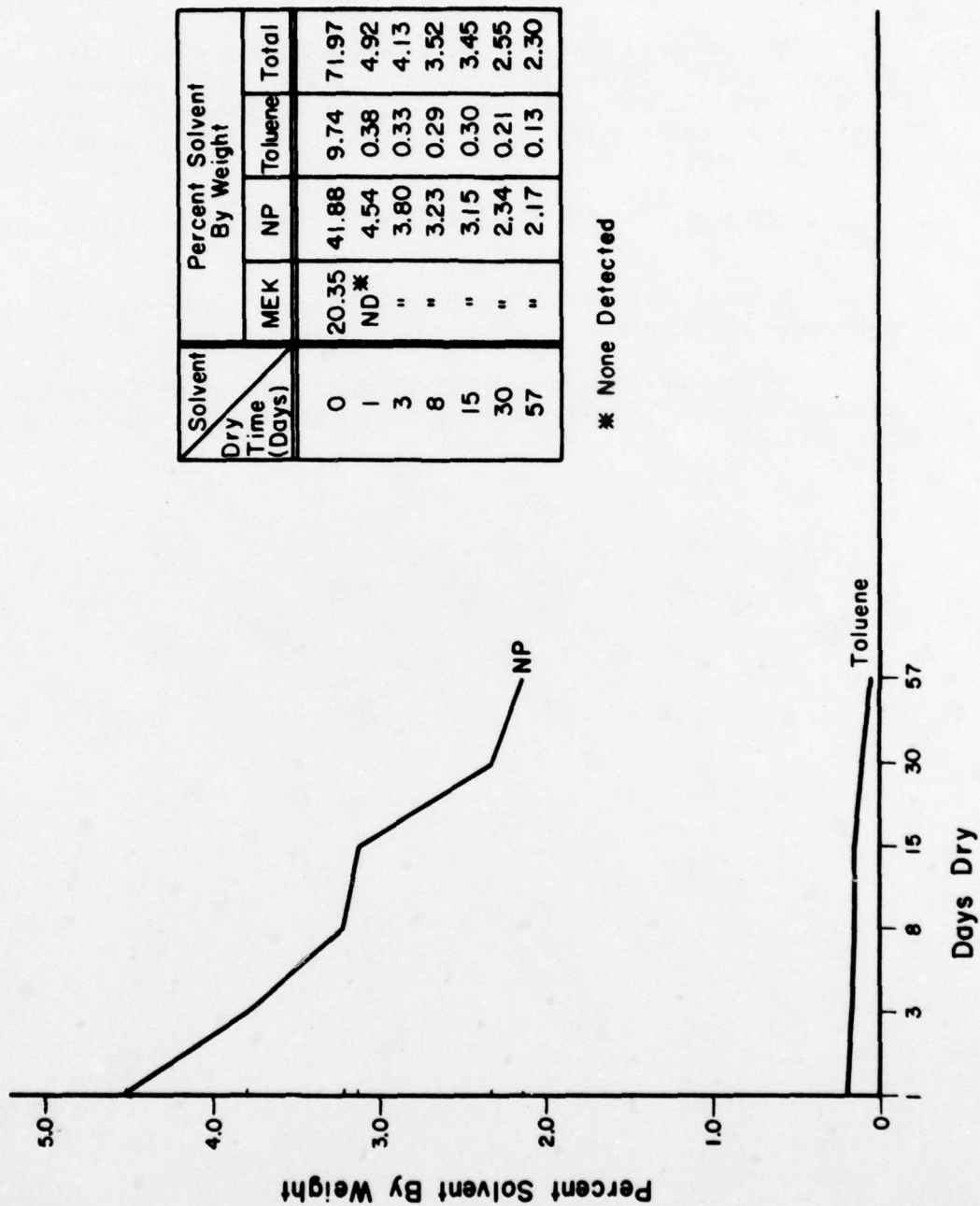
Xylene. Xylene shall conform to Federal Specification TT-X-916, Grade A (10 degree).

Thinner NP-10. Thinner NP-10 shall consist of 67 percent nitropropane solvent, 15 percent methyl ethyl ketone, and 18 percent toluene by volume.

APPENDIX B:

RETAINED SOLVENT TEST RESULTS

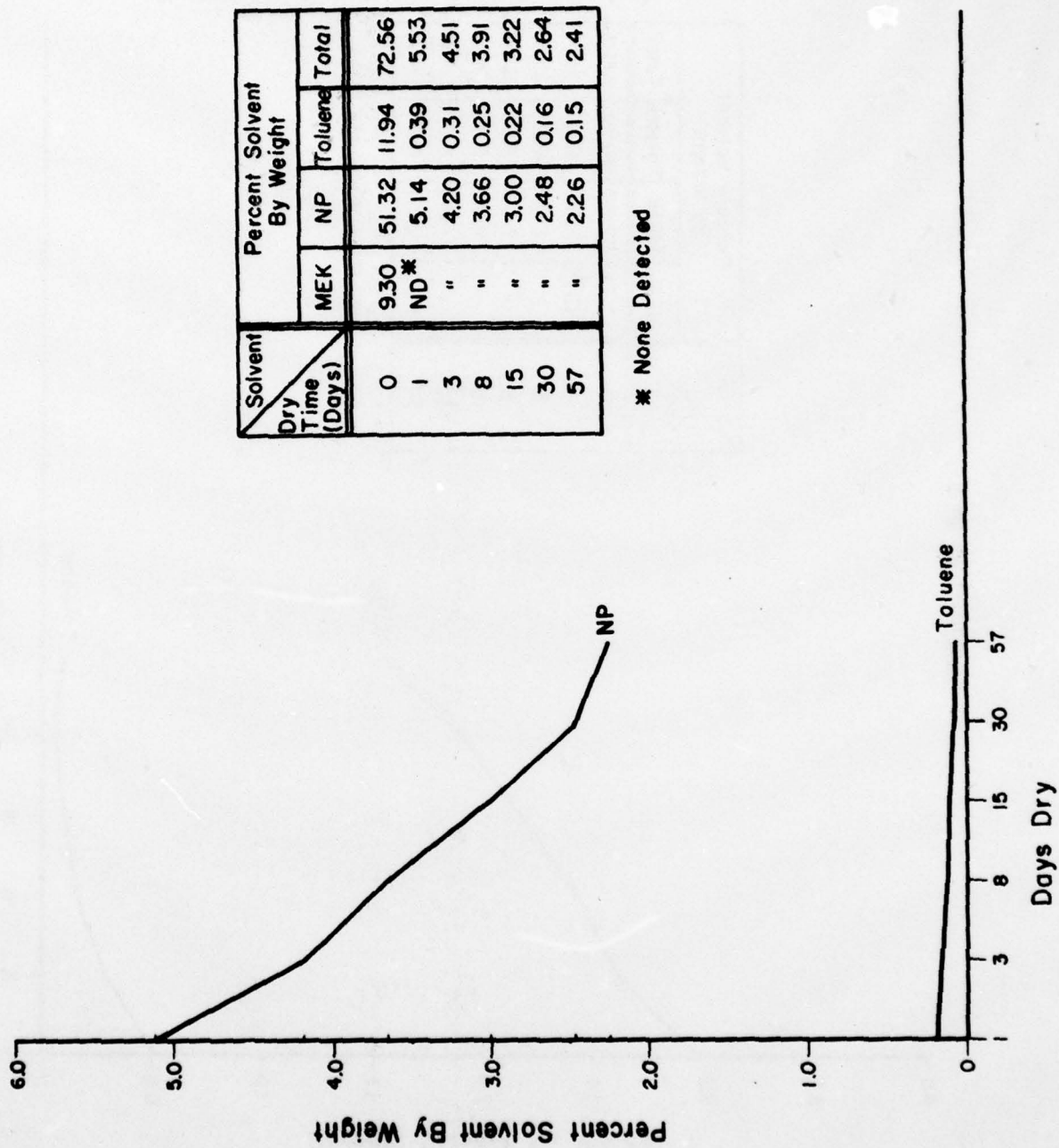
SAMPLE # 1



Solvent Dry Time (Days)	Percent Solvent By Weight			Total
	MEK	NP	Toluene	
0	20.35	41.88	9.74	71.97
1	ND*	4.54	0.38	4.92
3	"	3.80	0.33	4.13
8	"	3.23	0.29	3.52
15	"	3.15	0.30	3.45
30	"	2.34	0.21	2.55
57	"	2.17	0.13	2.30

* None Detected

SAMPLE #2



* None Detected

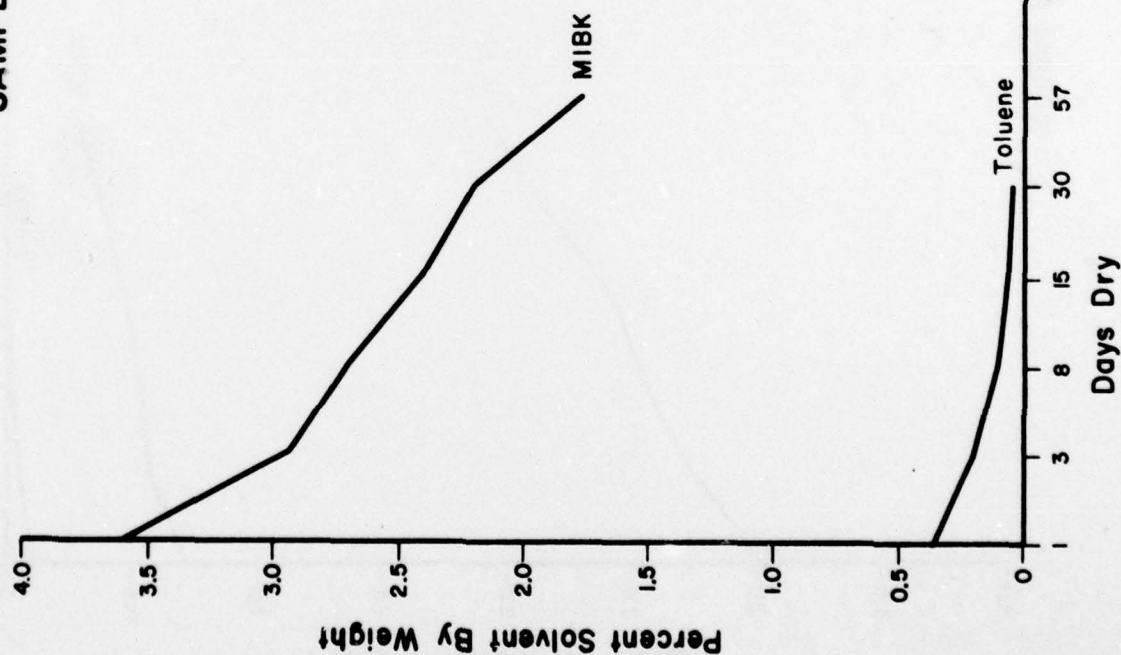
Solvent Dry Time (Days)	Percent Solvent By Weight		
	MEK	MIBK	Toluene
0	13.78	27.57	29.88
1	ND*	2.96	0.51
3	"	2.60	0.29
8	"	2.43	0.16
15	"	2.17	0.10
30	"	1.89	0.05
57	"	1.53	0.08

* None Detected

Solvent Dry Time (Days)	Percent Solvent By Weight			
	MEK	MIBK	Toluene	Total
0	13.78	27.57	29.88	71.23
1	ND*	2.96	0.51	3.47
3	"	2.60	0.29	2.89
8	"	2.43	0.16	2.59
15	"	2.17	0.10	2.27
30	"	1.89	0.05	1.94
57	"	1.53	0.08	1.61

* None Detected

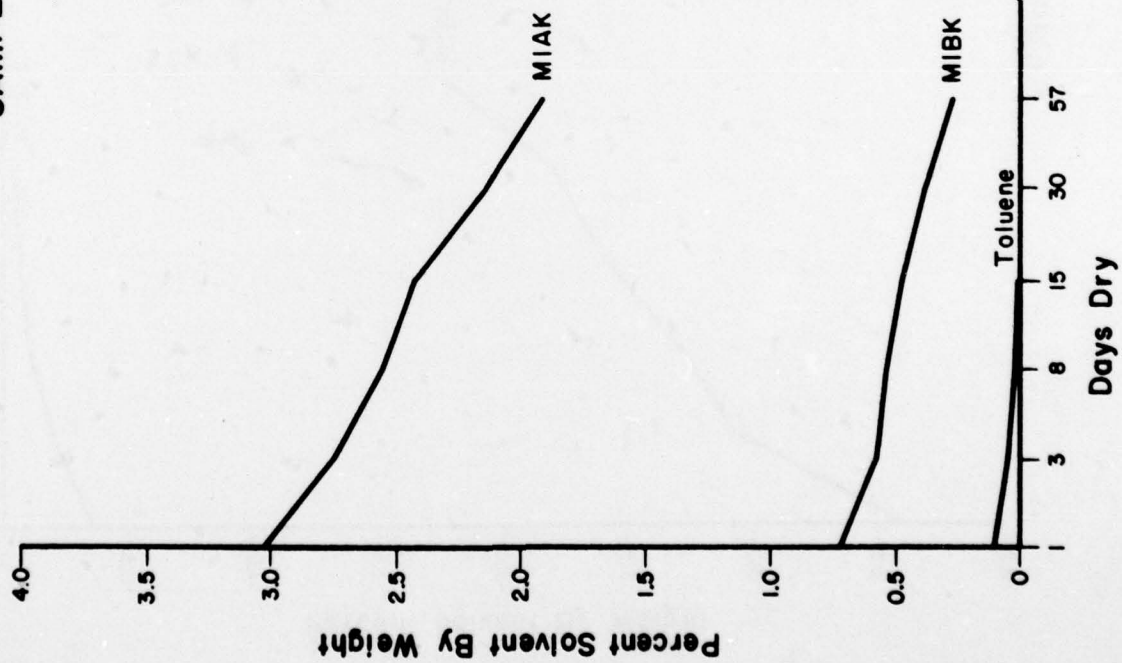
SAMPLE #4



Solvent Dry Time (Days)	Percent Solvent By Weight		
	MIBK	Toluene	Total
0	41.30	29.91	71.21
1	3.36	0.35	3.71
3	2.93	0.19	3.12
8	2.70	0.10	2.80
15	2.42	0.06	2.48
30	2.21	0.03	2.24
57	1.76	ND*	1.76

* None Detected

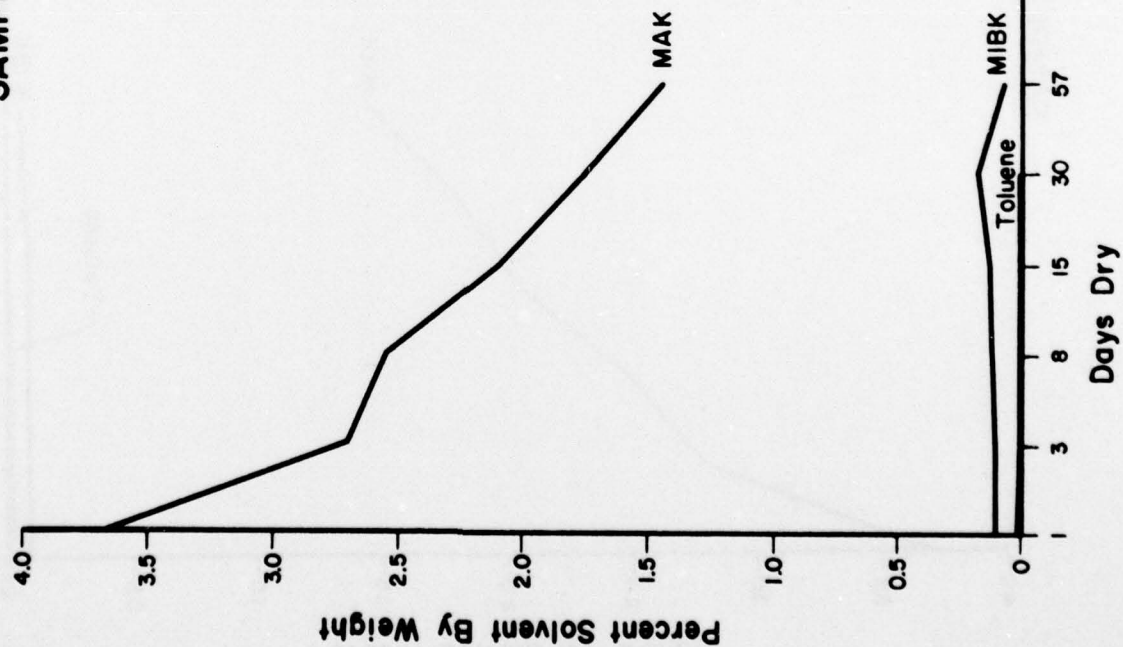
SAMPLE #5



Solvent Dry Time (Days)	Percent Solvent By Weight		
	MIBK	Toluene	Total
0	27.52	29.83	13.93
1	0.71	0.10	3.02
3	0.57	0.04	2.74
8	0.53	0.02	2.55
15	0.47	0.01	2.43
30	0.38	ND*	2.14
57	0.27	"	1.89

* None Detected

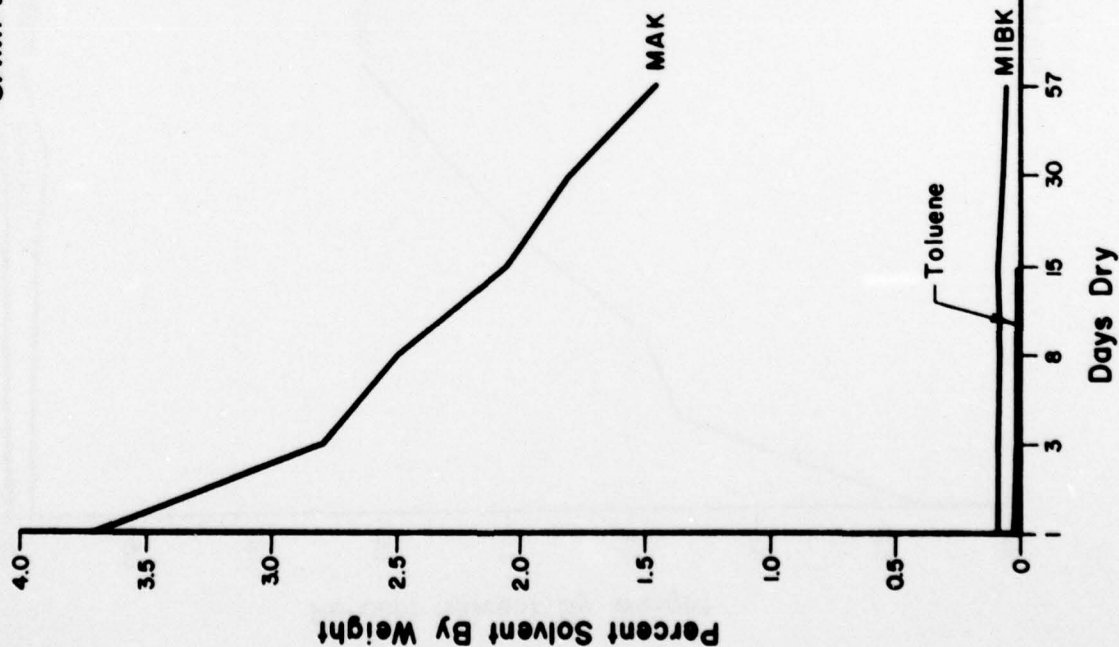
SAMPLE #6



Solvent Dry Time (Days)	Percent Solvent By Weight					
	MEK	MPK	MAK	Toluene	MIBK	Total
0	13.78	26.63	17.72	10.02	1.11	69.26
1	ND*	ND*	3.66	0.02	0.10	3.78
3	"	"	2.69	0.01	0.10	2.80
8	"	"	2.54	0.01	0.12	2.67
15	"	"	2.09	0.01	0.13	2.23
30	"	"	1.74	0.01	0.17	1.87
57	"	"	1.44	ND*	0.06	1.50

✖ None Detected

SAMPLE #7



Solvent Dry Time (Days)	Percent Solvent By Weight			
	MPK	MAK	Toluene	MIBK
0	39.86	17.71	10.01	1.71
1	ND*	3.71	0.02	0.08
3	"	2.78	0.01	0.08
8	"	2.49	0.01	0.07
15	"	2.06	0.01	0.08
30	"	1.80	ND*	0.06
57	"	1.45	"	0.05
				Total
				69.29
				3.80
				2.87
				2.57
				2.14
				1.86
				1.50

* None Detected

Solvent Dry Time (Days)	Percent Solvent By Weight				
	MPK	MAK	Toluene	MIBK	Total
0	26.38	32.15	9.92	1.10	69.55
1	ND*	3.87	ND*	0.02	3.89
3	"	2.94	"	0.03	2.97
8	"	2.49	"	0.04	2.53
15	"	2.34	"	0.03	2.37
30	"	1.86	"	0.03	1.89
57	"	1.10	"	0.03	1.13

* None Detected

*** None Detected**

Solvent Dry Time (Days)	Percent Solvent By Weight				
	MPK	MAK	Toluene	MIBK	Total
0	26.38	32.15	9.92	1.10	69.55
1	ND*	3.87	ND*	0.02	3.89
3	"	2.94	"	0.03	2.97
8	"	2.49	"	0.04	2.53
15	"	2.34	"	0.03	2.37
30	"	1.86	"	0.03	1.89
57	"	1.10	"	0.03	1.13

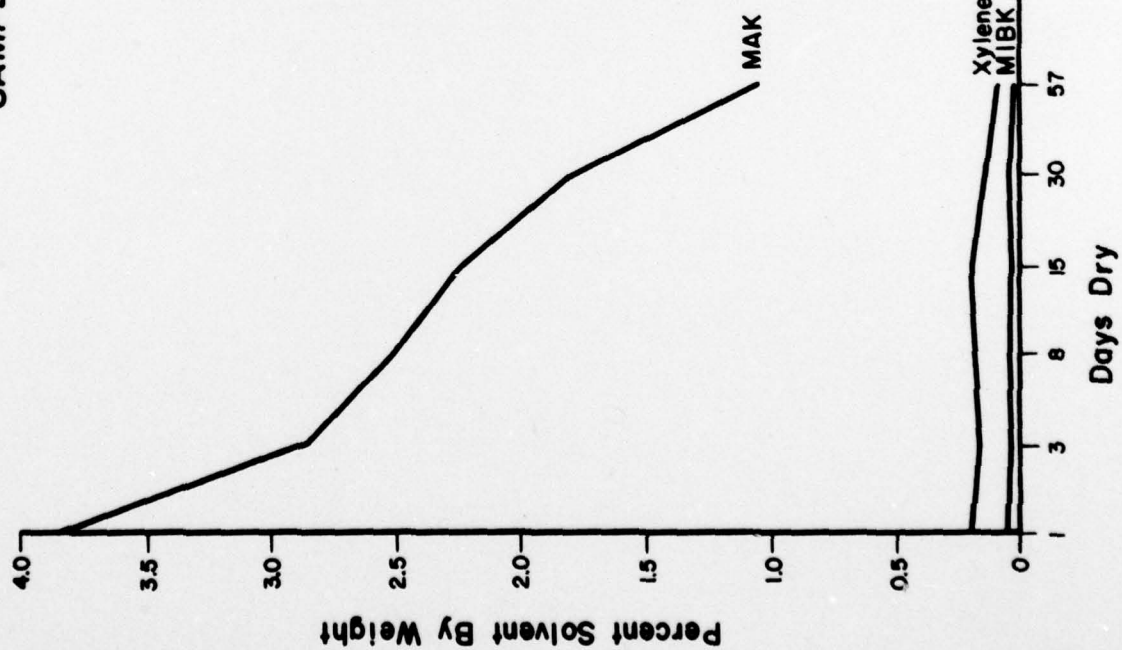
A line graph showing the percentage of solvent by weight remaining over time (Days Dry) for three solvents: MAK, MIBK, and Toluene. The y-axis is labeled 'Percent Solvent By Weight' and ranges from 0 to 4.0. The x-axis is labeled 'Days Dry' and ranges from 0 to 57. MAK shows a steady increase in solvent weight percentage over time, reaching approximately 1.2% at 57 days. MIBK and Toluene show very low solvent weight percentages, remaining near 0% throughout the 57-day period.

Solvent	Days Dry	Percent Solvent By Weight
MAK	0	0.0
MAK	3	0.2
MAK	8	0.4
MAK	15	0.6
MAK	30	0.9
MAK	57	1.2
MIBK	0	0.0
MIBK	3	0.0
MIBK	8	0.0
MIBK	15	0.0
MIBK	30	0.0
MIBK	57	0.0
Toluene	0	0.0
Toluene	3	0.0
Toluene	8	0.0
Toluene	15	0.0
Toluene	30	0.0
Toluene	57	0.0

Solvent Dry Time (Days)	Percent Solvent By Weight				
	MPK	MAK	Toluene	MIBK	Total
0	27.63	17.54	23.22	1.19	69.58
1	ND*	3.85	0.03	0.06	3.94
3	"	2.87	0.02	0.06	2.95
8	"	2.54	0.01	0.06	2.61
15	"	2.12	ND*	0.05	2.17
30	"	1.77	"	0.05	1.83
57	"	1.18	"	0.04	1.22

*** None Detected**

SAMPLE #10



Solvent Dry Time (Days)	Percent Solvent By Weight					
	MPK	MAK	Toluene	MIBK	Xylene	Total
0	26.36	18.98	9.92	1.10	13.22	69.58
1	ND*	3.85	ND*	0.04	0.19	4.07
3	"	2.84	"	0.03	0.16	3.03
8	"	2.51	"	0.04	0.18	2.73
15	"	2.24	"	0.03	0.19	2.47
30	"	1.79	"	0.04	0.13	1.96
57	"	1.03	"	0.02	0.08	1.13

* None Detected

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III. Series: U.S. Construction Engineering Research Laboratory. Special report ;
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